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CONTENTS

PROCEEDINGS OF THE ELISHA MITCHELL SCIENTIFIC SOCIETY..... 1

THE FOUNDATIONS OF SCIENCE.—*J. E. Mills*..... 3

5-BROM-2-AMINOBENZOIC ACID, A NEW PREPARATION.
Alvin S. Wheeler.....15

RAPID DETERMINATION OF OIL IN COTTONSEED PRODUCTS.
Chas. H. Herty, F. B. Stem, and M. Orr.....21

THE STABILITY OF ROSIN AT SLIGHTLY ELEVATED TEMPERATURES.
Chas. H. Herty and W. S. Dickson.....34

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PROCEEDINGS OF THE ELISHA MITCHELL SCIENTIFIC SOCIETY, NOVEMBER 1907 TO MARCH 1909

173RD MEETING, NOVEMBER 12, 1907

W. C. Coker: A Trip to Porto Rico. Illustrated with lantern slides and many botanical specimens.

174TH MEETING, JANUARY 10, 1908.

Dr. Styles, of the U. S. Marine Hospital Service, lectured on "The Condition of Cotton Mill Operatives in the South."

175TH MEETING, JANUARY 14, 1908.

H. V. Wilson: Some Phenomena of Coalescence and Regeneration in Sponges.

Chas. H. Herty: The Volatile Oils of *Pinus taeda* and *Pinus echinata*.

Chas. H. Herty: The Analytical Control of Cotton Seed Oil Manufacture.

176TH MEETING, FEBRUARY 12, 1908.

Collier Cobb: The Cause of Earthquakes in the Light of Recent Earthquake Action. Illustrated.

177TH MEETING, MARCH 31, 1908.

Archibald Henderson: Photography as a Fine Art.
A. S. Wheeler: Color Photography.

178TH MEETING, APRIL 28, 1908.

William Cain: Stresses in Masonry.
Wm. DeB. MacNider: Patholological Effect of Alcohol upon Animals.

BUSINESS MEETING, SEPTEMBER, 22, 1908.

The meeting was called to order by President Coker. The following officers were elected to serve for the ensuing year.
President: Archibald Henderson.
Vice-President: A. H. Patterson.
Permanent Secretary: F. P. Venable.
Recording Secretary: A. S. Wheeler.
Editorial Committee: W. C. Coker, Chairman; J. E. Latta, J. E. Mills.

179TH MEETING, OCTOBER 13, 1908.

H. N. Eaton: Results of the Microscopic Study of the Slate near Chapel Hill.
J. E. Mills: Chemical Energy.
H. V. Wilson: A Further Contribution on the Regenerative Power of Sponge Cells.

180TH MEETING, NOVEMBER 10, 1908.

Chas. H. Herty: Determination of Oil in Cotton Seed Products.
Collier Cobb: Geological Trip in Southern Europe in the Summer of 1908.

181ST MEETING, FEBRUARY 9, 1909.

W. C. Coker: A Visit to Luther Burbank in California. Illustrated with lantern slides and specimens of Burbank's productions.

A. S. WHEELER,
Recording Secretary.

THE FOUNDATIONS OF SCIENCE*

BY J. E. MILLS

I have thought for years and the conviction has grown stronger with each passing year, that much needless confusion existed among scientists regarding those broad principles upon which all science is founded. The student of science at first accepts the various definitions, laws, theories, and explanations, with which he is fed, with either an unquestioning obedience, or with a feeling of utter helplessness. Later he finds that his first learned definitions, laws, theories, and explanations, are incomplete or faulty or open to question on various grounds. What guide has he enabling him to climb above the confusion?

Chemists, certainly, have provided the chemists with none. There is an idea abroad among chemists that any discussions of the first and deepest principles of science are bound to be fruitless. Some chemists both act and talk as though they believed it a sin to look beyond and around the test tube which they hold in their hand. They imagine that they are thereby enabled to confine themselves to "facts" and they regard with more or less ill-concealed scorn the Brother chemist who refuses to be content with the fact but who insists on trying to obtain an explanation of the fact.

This attitude on the part of chemists, and it is true, I think, more particularly of American chemists, has to my mind been productive of a simply vast and untold amount of harm. For this attitude has discouraged the formal discussion, statement, and development, of the broad and underlying principles of science by scientists. We permit the philosopher to discuss these things but the philosopher too often lacks the knowledge and insight of

*Address of the President before the North Carolina Section of the American Chemical Society, Raleigh, N. C., Jan. 9th, 1909.

the scientist. We make a great mistake when we ignore, discourage, or ridicule the philosopher when he undertakes to consider the foundations of science. We should help him. Or rather we should absorb his knowledge. For the philosopher is trying to state certain broad principles underlying science and our ignorance of these first principles has led to a needless confusion, permitted many false explanations to become fastened on our science, and has turned many away from fruitful fields of investigation.

I have not overstated the case. I will give examples later of some of the many absurdities that are of every day occurence among us. But let us first consider briefly the origin of our knowledge.

There is a reality. No matter from what standpoint viewed, this much, we think, is admitted in all philosophies. There is, it seems to me, no more certain, and in fact no other ground upon which to rest such a conclusion than the "Cogito, ergo sum" of Descartes. I think, therefore I am. Our consciousness of the simplest mental act, of sensation, or of thought, or of volition, is sufficient to establish the fact. *Our first conclusion* is, that there is a reality. I think, therefore, at least *I* exist.

What more can we learn of this reality whose existence we are compelled to acknowledge ?

Our sensations are often deceptive and they may be always deceptive. Therefore we cannot trust them to give us any absolutely certain evidence about reality. Even were another sensation to be discovered we could never be sure that it would not likewise prove deceitful. The only thing of which I may be *absolutely* certain is that I exist. I think, therefore I exist. All else may be a dream. I may be the reality and the entire reality. There may be nothing but ideas, impressions. Or I may be but a part of one vast machine. We cannot with certainty condemn either the idealist or the materialist from this standpoint.

It has been pointed out that we have an equal right to declare, "Cogito, ergo cogitatio est", "I think, therefore thought is", which is true. But the conclusion that there is a non-ego as well as an ego—a thought as well as a thinker—does not necessarily follow. For when we come to consider the bottom of the matter the "thinker" and the "thought" may be one and the same thing.

True we have a very strong conviction that they are not the same thing, but this conviction is not absolute certainty. Since the actual nature both of the "ego" and of the "thought" is unknown, it becomes evident from the start that we cannot prove either their identity or their difference.

There is a reality—there absolute certainty ends. Upon this much all philosophers are practically agreed,

The second step in our search for knowledge is an assumption. Mathematics, physics, chemistry, all human knowledge, even reason itself, rests upon the following assumption:—

Likenesses and differences exist and can be recognized by us. If likenesses and differences did not exist and if you could not recognize these likenesses or differences you could not tell a friend from an enemy, you could not tell a good reason from a bad one, or a right conclusion from a wrong one.

Here science makes its start. The knowledge that we gain from day to day, or the knowledge that has come down to us from other laborers of other days, is derived mainly at least, and some believe that it is entirely derived, from a comparison of one object with another. We compare lengths, volumes, weights, colors, sounds, temperatures, and in short every property which will serve to distinguish two objects. If I wish to compare the lengths of two rivers, I cannot move the rivers side by side for comparison and so I compare each river with the length of a certain chain and draw my conclusion. But whether the comparison is direct or indirect, its nature is the same. No conclusion can be drawn where there is no common basis of comparison, where no common measuring stick exists. Thus no conclusion can be drawn from the comparison of a red light and a certain note of music.

The value of the comparison depends on the correctness of the conclusion drawn when the two objects are compared, and the correctness of the conclusion depends upon two things—the comparison and the mind. You must make the comparison carefully and you must trust the testimony of your mind as to the existence of the likeness or difference. I wish to emphasize the fact, that when the comparison has been carefully made, you are obliged to trust the testimony of your mind as to the existence of a likeness or difference. If you cannot trust your mind to give you a true

conclusion when the facts are accurately presented you cannot do business as a scientist or as a thinker.

The third point to which I wish to call your attention is the fact that the human mind holds certain beliefs with a tenacity which nothing can shake. Thus if I make the statement that "Two straight lines cannot enclose a space", you will agree that it is true. More than this you will agree that it is true of all straight lines. And you will agree immediately, and without argument, and with just as great a feeling of certainty after the first instant of thought as you will ever subsequently possess. Thinking about the matter, or seeing other straight lines, does not increase the first feeling of certainty which you had. Similarly you will agree at once that "Through a given point only one line can be drawn parallel to another given straight line". Also you will agree that "Nothing can both be and not be". You will agree that the "Whole is always greater than any one of its parts", and also that "The sums of equals are equal". Such statements are called axioms. Euclid called them *κοιναὶ ἐννοιαί*, or "common notions", and that is, perhaps, a better name.

Now why are we so certain of the truth of these and similar statements or axioms? Or as Sir J. S. Mill put the query "Why is a single instance, in some cases, sufficient for a complete induction, while in others, myriads of concurring instances, without a single exception, known or presumed, go such a very little way towards establishing a universal proposition?" Some believe that this intuitive conviction of the truth of certain statements is simply in the nature of a necessary condition of our thinking faculty. Others that this intuitive knowledge is in the nature of a free gift with our minds. Others that even such truths are the result of experience. And yet others, recognizing that our conviction of the truth of these statements cannot be wholly the result of our own experience, would have us attribute our belief to the experience of our ancestors, and if we are evolutionists, we may trace our feeling back to the monkey and the primordial germ.

Those philosophers who contend that all of our knowledge is derived from experience really seem to have the best of the argument. For we know that most of our knowledge is derived from experience. As we make wider generalizations the laws seem to us to possess a

greater certainty. What then is the advantage of resting even our most fundamental axioms on any other ground? Moreover inconceivability cannot be made a criterion of impossibility for man's ability to conceive of a thing of which he has had no suggestion is certainly very doubtful, if not admitted as an impossibility. And this fact alone, perhaps, explains our feeling of complete certainty when we give voice to fundamental axioms. If to all this Mr. Spencer's evolution theory of an inherited experience be added, there is an *a priori* conviction (feeling) that certain axioms are true so far as any particular individual is concerned. It is therefore impossible to dislodge by any argument these philosophers from their position.

On the other hand so far as the individual himself is concerned, it is certain, that there is a very great difference between the knowledge which he derives from his own direct experience, and the knowledge which he possesses apparently intuitively, and after all, it may be, that this intuitive knowledge is not the result of experience.

It is useless to follow the argument here. The scientist is not concerned primarily with the dispute as to the origin of these common notions which have been recognized and stated as axioms. From the standpoint of science the primary question is "Are these axioms true"? And if we say axioms are true then "What test can be adopted which will enable us to decide upon what an axiom really is?" We will define an axiom as *a statement whose truth our minds will not allow us to deny and of whose falsity our minds cannot even conceive.* If we accept the view of those who believe in an intuitive source of knowledge, then all axioms are equally true, and we must follow the intuition of our minds and accept those truths declared by our minds to be self-evident and impossible of contradiction. If on the other hand we agree with those men who think that all of our knowledge is derived from experience, or from the inherited experience of our ancestors, we cannot declare that any axioms are necessarily true. For accepting this point of view we must recollect that there may have been a mistake about the impressions all the way down the line from the primordial germ through the monkey to the man. And besides there may be experiences in store for us of a kind which it was

not the good fortune of our ancestors to have had. Thus each axiom becomes separately a subject of doubt.

We would, however, point out that no matter what our view as to the origin of our belief in the axioms, to deny the truth of the axioms is to deny the testimony of our own consciousness. And we must believe in the truth of the testimony of our minds if we would reason at all. This we have already made clear. You cannot deny the testimony of your consciousness and yet continue to reason, for the belief that your mind will give you a true judgement when the facts are accurately presented before it constitutes the very foundation of the reasoning process. If you agree with this idea then follow me to its conclusion.

Can you conceive of motion without something moved? Is not the idea of motion necessarily connected with the idea of a "something" that is moved? If your minds tell you that this idea is true, that it is necessarily true, and if you can not conceive of a contrary condition, then put this idea down as an axiom of science.

Again, can you conceive of a body acting where it is not across an absolutely empty intervening space? If you cannot, then let us put down as another axiom of science the fact, that a body cannot act where it is not, except through the intervention of another body.

Other axioms of science might be stated. For instance it might be stated that "The fundamental particle, or part, of matter can not be elastic". Many physicists would regard such a statement as beneath their notice. Yet it is nevertheless true that the property of elasticity implies a motion of certain portions of a body with reference to other portions of the same body, and so long as we have this relative motion of certain parts with reference to other parts we have not yet reached the final division limit of the body.

Personally I believe that the law of the conservation of matter and the law of the conservation of energy would both appear as axioms if we only understood more thoroughly the nature of matter and of energy. Likewise Newton's three laws of motion would doubtless appear to be axioms.

This morning I am not attempting to make a list of the axioms of science, nor shall I attempt here to trace out the consequences

that would follow from those already mentioned. The consequences are, I may say in passing, I think, both important and surprising. I do wish to emphasize this morning that chemistry and physics have as much right to these axioms as a mathematician has to the axioms of geometry. They rest upon precisely the same ground. They are common notions in which we are compelled to believe by the testimony of our minds. Moreover I wish to emphasize that a clear statement of these fundamental axioms and principles, and a clear understanding of them, would prevent many absurdities which act continually as a drawback to a greater advancement. I will give some concrete illustrations of my meaning.

1. How often do we find a statement that such and such a thing has been proved as a result of a mathematical investigation. Now a mathematical investigation can obtain as a result absolutely nothing that was not contained in the premises. The result may be given in a vastly more digested and convenient form. But nothing which was not contained, openly or concealed, in the premises, can be obtained in the conclusion. You cannot get more out of a mathematical process than you put in.

2. An investigation is often damned unjustly by a reviewer as being theoretical merely because it contains mathematics. A paper may be largely of a mathematical character and yet be in no sense more theoretical than is many a simple laboratory measurement or research.

3. Note the most advanced theory of our atom as given by the most advanced physicist of the present time, Sir J. J. Thompson. "Our atom therefore, we assume to be a sphere of positive electrification enclosing a number of negatively electrified corpuscles." If you look upon that idea as an assumption made to enable one to treat mathematically two opposing forces capable of producing equilibrium I have no fault to find. But if you are led away to worship that idea with a species of ancestor worship as representing the skeleton forefather of the ordinary atom your conscience should trouble you whether it does or not. The idea of having a shell or sphere made of nothing so that we may not be unduly troubled with questions about it is ingenious we admit, but does your mind consent to believe it true?

4. It is a known fact that if you pass an electric current through a solution of copper sulphate the copper is given off at one electrode and the SO_4 at the other, and one atom of copper is liberated for each atom of SO_4 and both atoms are liberated at the same time. Now to explain a certain change that takes place in the concentration of the solution around the electrodes Prof. Ostwald has attempted to show that the ions of Cu and of SO_4 can move with *different velocities* across the *same space* and yet be *given off at the same time*. And that very ingenious "explanation" has been copied in nearly every physics, and physical-chemical, and electrochemical text-book of the last ten years with a faith that passeth all understanding.

5. Another curious illustration of the absolute disregard of the testimony of our consciousness is the attempt to do away altogether with the idea of matter and to substitute energy as the kernel of all things. I cannot stop to discuss the reasons advanced for the change. I would only point out that whatever reasons may be advanced the position is an unsound one unless your mind can be made to give its consent. And neither your mind nor my mind can conceive of a world in which things do not occupy space.

I have a text-book on physical chemistry written by Prof. C. L. Speyers of Rutgers College in 1897 from this point of view. He tries hard to get along without the use of the terms, atom, molecule, and matter, I quote two paragraphs from his book:—

"In ordinary chemical language we say chemical reaction takes place between definite weights of matter. In our language we should say the relation between the intensities of the distance energies (gravity energies) of two or more collections reacting to form another definite collection is fixed.

When two substances combine in more than one proportion to form two or more bodies, in ordinary chemical language we should say the ratio of the varying substance to the fixed substance is in general more or less simple. In our language we should say when two collections are capable of uniting to form more than one new collection by their union, the ratio of the intensity of the gravity energy in the varying collection to the intensity of the gravity energy in the fixed collection is in general more or less simple."

The man is not crazy. He is simply carrying to an extreme

just the same ideas possessed by a good many other leading chemists.

6. Less curious, but perhaps even further wrong than the idea just mentioned, is the idea that we can talk about energy scientifically without making any assumptions regarding its nature. Now it was early recognized by the Greek and Roman philosophers, and I understand that much of their thought probably came from a far earlier Eastern philosophy, that if we considered matter to exist it could be divided. These parts could be divided again, and so on, until the very interesting question was presented, When, if ever, would the possibility of division stop? Chemists recognize as steps in this division the molecule, the atom, and perhaps the corpuscle or electron. Now many scientists seem to possess the idea that no such question necessarily arises regarding energy? They say in effect, "We know nothing of the nature of energy and why therefore discuss its divisibility or indivisibility? We make no assumptions concerning it one way or the other". I would answer this fallacy by an illustration. If I say one-third of the people in a car were killed in a certain railroad wreck my statement seems all right perhaps, but if there were ten people in the car then my statement is seen to be absurd. If you make no assumption about the nature of energy then you cannot treat energy mathematically. Euclid overlooked a certain axiom concerning space which modern geometricians recognize as being necessary, namely, that "Space is continuous". Modern scientists should not overlook the assumptions made regarding energy. The continuity or the divisibility of energy is a problem exactly on a par with the problem as to the continuity or divisibility of matter?

7. Physicists and chemists do not stand alone in their attempts to thrust aside the testimony of their minds as to one concept, and to accept at the very same time the conclusions of the very same mind regarding other things. Thus there are a good many mathematicians who speak of the possibility of a space of "n" dimensions. Now we can conceive of a point, of a line, of a surface, and of a solid. And we understand quite readily that one unknown quantity might be used to fix the position of a point on a line, that two might be used to define or to represent points on a plane surface, and that similarly three would serve to indicate

the points in a solid. We understand quite as readily that an equation with four unknown quantities, or with five unknown quantities, can be constructed, and that this equation would likewise represent certain relations between the unknown quantities. But to declare that this equation also might represent space is as meaningless as it would be to declare that it represents time. Such a declaration is certainly contrary to the testimony of the very same mind that we have used as our guide in the mathematical reasoning, and to accept its conclusions at one point and reject them at another is an entirely indefensible position.

8. Non-Euclidean geometry is an illustration of the same method of proceedure. Gauss undertook to investigate mathematically the properties which a surface must possess in order to permit figures to be moved about upon the surface without altering the shape or the size of the figures. He found that there were three classes of such surfaces. A plane surface, the surface of a sphere and the surface of a figure which became known as the pseudo-sphere. Lobatchewsky, a pupil of Gauss, undertook a similar investigation regarding space. What properties must space possess in order that bodies could be moved about in the space from one part of the space to another without altering the shape or the size of the bodies? He found similarly three classes of equations, one representing the ordinary Euclidean, or plane space, the others corresponding to the sphere and the pseudo-sphere of the investigation carried on by Gauss. Many wonderful results followed from these investigations and I am not intending to detract from the value of that work, or of similar work. But one of the results arrived at was, that in "plane" space only, was the following axiom true:—"Through a given point only one straight line can be drawn parallel to a given straight line." In both the "spherical" and the "pseudo-spherical" space this axiom was untrue. Now I do not question the correctness of the work or of the equations. But I do question that we have a right, because of them, to cast aside the unmistakably clear belief in the above axiom forced upon us by our minds. Whatever the other equations may represent, or whatever their meaning, we have no right to violate the testimony of the mind that derived them, and declare that they represent space.

I have not time this morning to give further illustrations of my statement that the lack of knowledge of the first principles of science, as shown by scientists, is the cause of much needless confusion. If I were to point out individual errors due to this cause that have come to my attention I would occupy the entire day. These errors are caused, not by any failure of our minds to give us a true judgement, but by our own failure to read carefully the answer which our mind has given. If in the last analysis the testimony of consciousness cannot be trusted we had just as well give up the search for truth. We cannot hope to attain to any absolute knowledge or full conception of any of the more elementary ideas such as time, space, matter, motion, or energy. But we may attain to a partial knowledge of these ideas and this partial knowledge, we trust, may represent the reality truly, so far as it represents it at all. And in attempting to attain this partial knowledge, if one goes directly contrary to the testimony of one's mind as to the possibility or impossibility of a conception, one should not forget that the process of denying the truth of the testimony of consciousness once begun, can be as legitimately extended to an absolute agnosticism. Must be so extended, if one is consistent. If a scientist should wish to introduce into his science an idea which is contrary to the conception of things imposed upon him by his mind, let him do so. But he should himself understand, and he should make clear to others, that he has not only contradicted one particular "necessary concept" of his mind, but that *in so doing he has inaugurated a process which could consistently be extended so as to undermine his whole structure.* A scientist may state that matter may be but a center of force, and we have no fault to find. But he has no right, in the next breath, to swear by the result of a mathematical investigation which involves axioms resting upon the same ground as the one that he has just contradicted. If greater attention were given to the foundations of science doubtless some phenomena would remain "unexplained" but some very confusing ideas would be banished from the domain of science.

In conclusion I would ask those who are here today, if your mind has agreed with the ideas presented, to endeavor so far as it may be in your power, to bring about among chemists a clearer

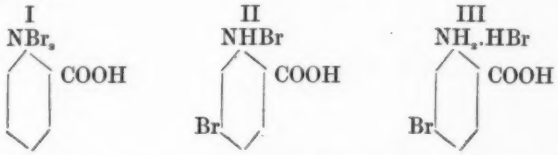
recognition of the service which a discussion of the first principles of science would render to chemistry. The attitude of chemists and of other scientists, is today so pronounced in opposition to such discussion that it is exceedingly rare to find a text-book which even alludes to the subject. Nowhere today, does a scientific student, who has not read or studied philosophy as well, obtain an idea of the basis of thought, or of mathematics, or even of science itself. Disagree with me you may concerning some of the particular statements that I have made this morning. But your very disagreement itself speaks in favor of a discussion of these first principles in order that we may the sooner clear away the misunderstandings which exist. At present one can hardly attempt their discussion without losing in some measure the respect of his fellow scientists. My brethren these things ought not so to be.

CHAPEL HILL, N. C.

5-BROM-2-AMINOBENZOIC ACID, A NEW PREPARATION

ALVIN S. WHEELER

The original object of this investigation was to convert trichlorethylidene-o-aminobenzoic acid[1] into a compound containing an asymmetric carbon atom by means of bromine or hydrobromic acid, thus $CCl_3CH:NC_6H_4COOH=CCl_3CHBrNBrC_6H_4COOH$. A glacial acetic acid solution of the unsaturated compound absorbs bromine instantly in the cold with the immediate formation of a white precipitate. A Carius determination showed a marked deficiency of halogen and displacement of the bromine in the silver precipitate proved the entire absence of chlorine in the substance. Further proof that the chloral residue was split off was found in an examination of of the acetic acid filtrate. This was made alkaline, then acid with tartaric acid and distilled in steam. A large quantity of chloroform passed over with steam. The product was therefore a simpler derivative of anthranilic acid than the one looked for. Since the break occurs at the double bond, it might be a nitrogen bromide I or II, or the hydrobromide of a bromanthranilic acid III.



Difficulty was experienced in obtaining products of the same melting point, different solvents giving slightly varying results. While none of the reactions of the nitrogen halides as indicated by Chattaway and Orton[2] could be obtained, it was thought this

[1] Niementowski and Orzechowski, Ber. 28,2812; also Wheeler, Jr. Am. Chem. Soc. 30,139.

[2] Chattaway and Orton, J. Chem. Soc. 75,1046.

might be due to the insolubility of the compound. But the idea of a nitrogen bromide was abandoned when it was observed that cold water immediately and completely removed one molecule of hydrobromic acid, showing the substance to be the salt of bromaminobenzoic acid. It is still hoped to isolate in some way the nitrogen bromide which must certainly be the first product of the reaction. The case is an especially interesting one because it is quite unique in that the substance employed contains the grouping $-N=X$ and so seems to offer an unusually good chance to form a nitrogen halide.

It was next observed that direct bromination of anthranilic acid in glacial acetic acid solution gave a similarly large yield of the bromaminobenzoic acid hydrobromide. The reaction therefore offers a new method of preparing this acid far superior in point of time and convenience to the method of Alt[1] which has been recently used by Bogert and Hand.[2] This old method consists in brominating o-acettoluide, oxidizing the brom-o-acettoluide with permanganate and saponifying the bromacetanthranilic acid. The method requires as many days as this new method requires hours. A comparison of the bromination of the chloral-anthranilic acid and of anthranilic acid shows that the chloral residue has some influence on the course of the reaction.

Bromination of chloral-anthranilic acid. 5-Brom-2-aminobenzoic acid hydrobromide, $C_6H_3COOHBrNH_2.HBr$. A saturated solution of chloral-anthranilic acid (m. 152°) was made by dissolving 13.3 grams in 100 cc glacial acetic acid by warming and then cooling nearly to the room temperature. Bromine, 8 grams, was added drop by drop with stirring. Crystals began to form before half of the bromine was added. Finally a very thick pulp was obtained, the temperature having risen to about 45.° The proportions of the materials were 1 molecule of acid and 2 atoms of bromine. The precipitate was filtered off and washed several times with benzene. The compound so obtained is snow white and nearly pure. It gives a melting point of 238-40°, decomposing and becoming

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[1] Alt, Ber. 22, 1645, (1889).

[2] Bogert and Hand, Jr. Amer. Chem. Soc. 27,1476 (1905).

an indigo blue liquid immediately above this temperature. The yield was equal to the weight of the chloral-anthranilic acid used or 91 per cent of the theoretical. Analysis: calculated for $C_7H_7O_2NBr_2$, Br 53.86, found, Br 53.40, 54.16, 53.45.

The hydrobromide is broken down at once by cold water into the free amine and hydrobromic acid which accounts for difficulties arising in recrystallizing from solvents not especially dried. It is insoluble in ether, benzene, ligroin, carbon tetrachloride and chloroform, slightly soluble when these solvents are hot. It is insoluble in cold glacial acetic acid but soluble in about 150 parts when boiling, separating out in needles. It is rather soluble in cold absolute alcohol, soluble in 6 parts when boiling, crystallizing out in needles grouped in rosettes.

5-Brom-2-aminobenzoic acid. $C_6H_3COOHBrNH_2$. Although the acid is set free from its hydrobromide by cold water, hot water was used in order to effect a recrystallization of the acid simultaneously. The acid was found to agree in all its properties with the acid as described by Alt and later by Bogert and Hand except in one particular. Alt gives the melting point as 211–211.5° (uncor) while Bogert and Hand give 219–220° (cor.). Our acid melts at 215–216° (uncor) or 218–219° (cor), in practical agreement with Bogert and Hand. We have observed further that a partial decomposition takes place. As Alt has stated an aqueous solution of the acid turns violet in the sunlight and a solution of its ammonium salt gives colored precipitates with certain other salts. Its acetyl derivative consists of splendid prisms, melting at 218–220°. Alt gives 214–215°. Analysis: calculated for $C_9H_8O_3NBr$, Br 31.01: found, Br 31.62. The position of the bromine atom was positively located by conversion of the acid into m-brombenzoic acid by the diazo reaction.

Bromination of Chloral-di-anthranilic Acid. This acid is described by Niementowski[1] and by Wheeler and Dickson[2]. 2.0 grams of chloral-di-anthranilic acid (1 mol) were dissolved in 30 cc glacial acetic acid and treated with 1.6 grams (2 mols) bro-

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[1] Niementowski, Ber. 34,3898 (1902).

[2] Wheeler and Dickson, this Journal, 30,140 (1908).

mine. The abundant white precipitate was worked up as in the previous case. The yield was 2.7 grams or 91.5 per cent of the theoretical. The melting point was 238-239° with decomposition to an indigo blue liquid. Cold water removed one molecule of hydrobromic acid. The residue was recrystallized from water, forming needles melting at 215-216° with partial decomposition. The reaction is therefore identical with bromination of chloral-mono-anthranilic acid.

Rapid Preparation of 5-Brom-2-aminobenzoic Acid. First Method, with Use of Chloral. Since it is immaterial whether the chloral-mono-anthranilic acid or the chloral-di-anthranilic acid is used in the bromination, the products of the action of chloral upon anthranilic acid need not be separated. 25 grams of anthranilic acid are rubbed up in a mortar to a powder, 27.5 grams of chloral are added and the mixture is rapidly triturated a few minutes. The mass partially liquifies with the development of heat and then becomes quite hard. It is at once dissolved in 350 cc glacial acetic acid by warming and then cooled to 16°. Bromine, 29.4 grams, is added slowly enough to keep the temperature from rising above 16°, the beaker being surrounded by cold water. The precipitate is filtered off and washed with benzene. It melts at 239-240° and weighs 51 grams or 94 per cent of the theoretical. Its conversion into the free acid and its purification were effected as follows. 5 grams of the hydrobromide were boiled up in 250 cc water three times, filtering hot with suction into a flask standing in boiling water. The filtrates were cooled by surrounding with ice water and then filtered. Extract I, m. 215-216°, weight 1.35 gram; extract II, m. 215-216°, weight, 0.95 gram; extract III, m. 208-210,° weight, 0.35 gram. Insoluble residue none. By adding 3 volumes of water to the glacial acetic acid filtrate, a precipitate was obtained, melting at 185-200° and weighing 0.3 gram. Analysis of this product showed it to be a tribromaminobenzoic acid. The bromine (29.4 g) is accounted for as follows: 2 g lost in the original preparation, 14.7 g are lost as HBr, 10.4 g appear in the 5-brom-2-aminobenzoic acid, 1.9 g appear in the tribromaminobenzoic acid. The balance, 1.4 g, disappear in the mother liquors. Recrystallization of extract I from toluene effected no rise in melting point. In some preparations the first extracts

were not quite so pure and one recrystallization from toluene was necessary. At no time was boneblack necessary as in the old method. Analysis: calculated for $C_7H_6O_2NBr$, Br 37.04; found, Br 37.39.

Second Method. Direct Bromination of Anthranilic Acid. (With W. M. Oates.) 25 grams anthranilic acid were dissolved in 250 cc glacial acetic acid and cooled to 15-16°. Bromine, 29.4 grams, was added slowly enough to keep the temperature from rising. Further procedure was the same as described above. The product melted at 236-238° and weighed 51.5 grams which is 95.3 per cent of the theoretical. Conversion and purification were carried out as already described. 5 grams of the hydrobromide gave the following results: extract I, m. 212-214°, weight, 1.4 g; extract II, m. 212-214°, weight, 0.8 g; extract III, m. 203-205°, weight, 0.3 g; extract IV, m. 226-232,° weight, 0.1 g. Undissolved residue, m. 228-230°, weight, 0.4 g. The first extract was recrystallized from toluene and analyzed. Analysis: calculated for $C_7H_6O_2NBr$, Br 37.04; found, Br 37.44, 37.65. The undissolved residue, m. 228-230°, was also analyzed. Analysis: calculated for $C_7H_5O_2NBr_2$, Br 54.24; found, Br 52.18. It was therefore a dibrom derivative of anthranilic acid. The bromine, 29.4 grams, is accounted for as follows: 1.7 g lost in the original preparation; 13.8 g lost as HBr, 9.5 g appear in the 5-brom-2-aminobenzoic acid; 2.8 g appear in the dibrom anthranilic acid and the balance, 1.6 g, are lost in the mother liquors. The acetic acid filtrate in this method of preparation did not yield any precipitate on the addition of water. A comparison of results in the two methods is as follows:

I	II
Mainly 5-brom-2-aminobenzoic acid	Similar amount
Small amount tribrom acid	No tribrom acid
No dibrom acid	Small amount dibrom acid

m-Brombenzoic Acid. This acid was prepared in order to locate positively the bromine atom in the bromaminobenzoic acid described in this paper. It proved to be a convenient method of preparation because the product is so quickly purified, which can

not be said of the product of the action of bromine on benzoic acid. 5.0 grams 5-brom-2-aminobenzoic acid were dissoved in 50 cc absolute alcohol. After the addition of 5 g H_2SO_4, con., the solution was brought to boiling and 2.5 g $NaNO_2$ dissolved in a very little water were added. The solution was filtered from the sodium sulphate and added to a large volume of water. The pinkish colored precipitate was filtered off and dried. It showed a melting point of 145-147° and weighed 3.82 g or 82 per cent of the theoretical. On recrystallizing from boiling water a very small amount of a dark red substance was gotten rid of and the m-brombenzoic acid was obtained in pure condition, m. 154°. Analysis: calculated for $C_7H_5O_2Br$, Br 39.80; found, Br 39.37.

University of North Carolina,
Chapel Hill, N. C.
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RAPID DETERMINATION OF OIL IN COTTONSEED PRODUCTS*

BY CHAS. H. HERTY, F. B. STEM AND M. ORR

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In the manufacture of cottonseed oil the delinted seeds are cut and the meats or kernels separated mechanically from the hulls. The meats are then mashed between heavy rollers, cooked in steam jacketed vessels, and the oil expressed by hydraulic pressure. The determination of oil in the hulls gives a measure of the completeness of the separation of the meats from the hulls, while the oil content of the meal affords a control of the character of the press work. It would seem, therefore, that a prompt and constant knowledge of the oil content of the hulls and meal would be indispensable in the operation of a mill. However desirable, this is by no means the case in actual practice. Two explanations suggest themselves: first, the organization of the industry; second, the method of analysis employed. The erection of many small mills is made necessary by the bulky character of the cottonseed, with its consequent cost of transportation, and by the impossibility of storing large quantities of seed for any great length of time without marked deterioration. The output of many of these small mills is not sufficient to justify the employment of a trained chemist, and the time required for obtaining an analysis from a distant laboratory largely discounts the value of the knowledge gained, except in extreme cases. But even if a laboratory is close at hand, or in the mill itself, five or six hours must elapse before the result of the analysis reaches the mill from the laboratory, this on account of the method of analysis employed. A more general resort to chemical control could be

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expected, therefore, if a simple and rapid method of analysis were available. The present investigation has been undertaken in the hope of filling this need. Furthermore, the purchase of cottonseed by the mills is conducted regardless of the oil content of the seed. It is hoped that the rapid method here described may be of some service in this important matter.

OBJECTIONS TO PRESENT METHOD

The method of analysis usually employed consists in extracting the oil from a sample in a Soxhlet extractor with redistilled, low-boiling petroleum ether, evaporating the extractive and weighing the residual oil.

The objections to the method are:

First, the extraction must be conducted at a relatively low temperature, this being determined by the temperature of the condensed extractive dropping upon the sample from the condenser above.

Second, in the Soxhlet apparatus it is necessary not only to completely extract the oil but to transfer it completely to the flask below by repeated siphonings.

Third, considerable time is consumed in the complete evaporation of the extractive.

Fourth, the limitation to a comparatively small sample of the substance.

Fifth, the danger from fire.

Sixth, the necessity of using rather expensive apparatus and the need of a supply of running water for the condensers.

Seventh, the necessity of employing a trained chemist to conduct the operations.

PROPOSED METHOD

It is believed that all of these objections are met in the method here proposed. It consists in digesting a definite and relatively large quantity of the substance with a definite volume of carbon tetrachloride in a loosely stoppered Erlenmeyer flask for 15 minutes at a temperature of approximately 60° C., cooling to room temperature, shaking thoroughly, filtering and determining the specific gravity and temperature of the filtered extract. From

these observations the per cent. of oil is calculated. Detailed directions for the analysis of the several products follow:

Meal.—Forty grams of the thoroughly mixed sample of meal are transferred to a 250 cc. Erlenmeyer flask, which is then placed in a water bath previously heated to approximately 60° C. After heating the flask about 2 minutes 100 cc. of carbon tetrachloride are poured gently upon the meal through a funnel reaching almost to the surface of the meal. The flask is lowered in the water until the surface of the carbon tetrachloride is lower than that of the water, a cork loosely inserted and the extraction allowed to proceed. Slight variations in the temperature of the water bath do not affect the accuracy of the determination. The minimum time for the extraction is 15 minutes, it may be longer. When the extraction is completed the flask is cooled to room temperature, thoroughly shaken and the contents strained through wire gauze on to a folded filter, the gauze being squeezed to insure sufficient filtrate. The clear extract is caught in a specific gravity tube (5 inches by 1 inch test-tube on foot). This is filled to within one inch of the top, then tightly corked and placed in a vessel containing water at room temperature. After standing at least 10 minutes the tube containing the extract is placed in a 500 cc. plain beaker containing sufficient clear water, at room temperature, to reach the level of the extract in the tube. By this means too rapid change in the temperature of the extract is avoided. The cork is then removed, the specific gravity of the extract determined by the Westphal balance, and the temperature accurately noted from the thermometer placed within the plummet of the balance. Add or subtract the constant for the carbon tetrachloride in use and subtract the corrected specific gravity of the extract from that of the carbon tetrachloride shown in Table 6 at the same temperature. This difference is divided by 0.00286. The result is the per cent. of oil.

Hulls.—After removing whole seed from the sample of hulls, 40 grams are placed in an Erlenmeyer flask and slightly packed with a glass rod. This is necessary to insure complete covering of the bulky hulls by the extractive. 100 cc. of carbon tetrachloride are added. The rest of the determination is identical with that for meal, except that the gauze strainer is not necessary in filtering.

Meats. The method is the same as for meal except that 10 grams of meats are used with 100 cc. of carbon tetrachloride. The per cent. of oil found is multiplied by four.

Seed.—The sample of seed is thoroughly pounded in an iron mortar and the mass passed through a meat chopper. Forty grams are used with 200 cc. of carbon tetrachloride. The method is the same as for meal except that the per cent. of oil found is multiplied by two.

Experimental

THE EXTRACTION

The well known solvent power of carbon tetrachloride for fats, its relatively high boiling point, its low heat of vaporization and its non-combustibility, naturally suggested this substance as a substitute for gasoline and ether as the extractive. A P. Bryant,[1] using a Knorr extractor, demonstrated the rapidity of extraction of fats in foods and feedstuffs by carbon tetrachloride. But he proposed merely the substitution of carbon tetrachloride for ether or carbon bisulphide in the usual method of extraction and evaporation of the extractive.

In the following experiments the determinations were carried out as follows: five grams of a substance whose oil content had been determined by the Soxhlet method were transferred to a 500 cc. Erlenmeyer flask, 50 cc. of gasoline or carbon tetrachloride added, and the extraction allowed to proceed at room temperature and without shaking, in order to avoid the sticking of the solid particles to the walls of the flask above the extractive. After standing a definite time the flask was thoroughly shaken in order to insure uniformity of the extract. This was then filtered through a dry filter paper. The amount of dissolved oil was determined in 25 cc. of the filtrate by distilling off the extract in a hot air bath, the temperature being quickly raised to 140° C. during the last stages of the evaporation. The last traces of the extractive were removed from the distillation flask by blowing quickly into it through a glass tube inserted almost to the oily layer. From the weight of the oil the per cent. was calculated by multi-

1 *J. Am. Chem. Soc.*, 26, 568.

plying by one hundred and dividing the product by two and a half.

Cottonseed Meal.—7.29 per cent. oil by Soxhlet extraction with gasoline.

TABLE 1

Time of extraction.	Per cent. oil with gasoline.	Per cent. oil with carbon tetrachloride.
7½ minutes	6.72	2.84
15 "	7.32	7.10
30 "	7.34	7.22
60 "	7.28	7.30
120 "	7.32	7.46

To determine the influence of temperature upon the rate of extraction by carbon tetrachloride, a series of experiments was carried out as above except that the Erlenmeyer flasks were placed in a water bath heated to 50° C. The results follow:

TABLE 2

Time	Per cent. of oil.
3¾ minutes	7.08
7½ "	7.44
15 "	7.38

The marked increase in the extraction at 50° in 7½ minutes as compared with that at room temperature for the same time led to the hope that the time for complete extraction could be still further diminished by carrying out the determination near the boiling out the determination near the boiling point of carbon tetrachloride. This hope was fully justified, for by making an extraction at 70° for 3¾ minutes, the percentage of oil was found to be 7.30.

Cottonseed Meats.—30.27 per cent. oil by Soxhlet extraction.

TABLE 3

Time.	Temperature.	Per cent. of oil.
30 minutes	25°	29.22
30 "	35°	29.50
30 "	50°	30.38
30 "	70°	30.62

From these determinations it is evident that the time-consuming extraction with the Soxhlet apparatus is unnecessary and that an accurate determination of the oil in these products can be made with simple apparatus, in a much shorter time and with no danger from fire.

RAPID DETERMINATION OF OIL IN EXTRACT

In order to simplify and hasten the determination of oil in the extract experiments were begun on the lowering of the specific gravity of the carbon tetrachloride by the dissolved oil. The great difference in the specific gravity of carbon tetrachloride (1.62) and of cottonseed oil (0.92) gave a reasonable hope of success. In order to avoid possible difference in the specific gravity of the crude oil obtained from the presses and that left in the meal the experiments were carried out on the samples of meal whose oil content was determined in the usual manner by the Soxhlet extractor. The simplicity of the Westphal balance suggested this method of determining the specific gravity. Comparison of the thermemeter on the plummet of the balance used in the investigation with a standard thermometer showed it to be accurate between 15° and 30°.

A preliminary test, using 5 grams of meal and 50 cc. of carbon tetrachloride, as in the above experiments on the rate of extraction, showed that the method would probably be accurate to 1 per cent. The first step, therefore, was to determine the minimum time and temperature for extractions of larger proportions of meal, the volume of carbon tetrachloride being kept constant. A sample of cottonseed meal was passed through a 20-mesh sieve, and thoroughly mixed. In two portions of ten grams each the oil was determined by the Soxhlet method, using redistilled gasoline as the extractive in one case and chemically pure carbon tetrachloride in the other. With the former the percentage was found to be 8.68; with the latter 8.67.

With this meal extractions were made according to the method outlined above. In each case allowance was made for the constantly increasing volume of oil in the extract. The results follow:

TABLE 4

No.	Grams of meal.	cc. of carbon tetrachloride.	Time (minutes).	Temperature.	Per cent. of oil I	II
1	10	50	5	70°	8.67	...
2	15	50	5	70°	8.58	...
3	20	50	5	70°	8.60	8.63
4	25	50	5	70°	8.18	...
5	30	50	5	70°	8.56	...

From the per cent. found in No. 5 it is evident that the low result in No. 4 was due to analytical error.

The use of 30 or even 25 grams of meal was found to be impracticable, as it was found to be impossible to secure sufficient filtrate for use with the Westphal balance. But with 20 grams sufficient filtrate could be obtained by using a strainer of brass or iron gauze and squeezing the extract on to a crimped filter. In order to avoid possible error in the specific gravity determinations due to the small amount of filtrate the proportions were increased to 30 grams of meal and 75 cc. of carbon tetrachloride. For the specific gravity determination a test-tube-on-foot 5 inches long and 1 inch in diameter was used. A magnifying glass was used in reading the thermometer on the plummet of the balance.

TABLE 5

SPECIFIC GRAVITY OF CARBON TETRACHLORIDE.

Temperature.	Specific gravity.
15°	1.6041
20°	1.5951
25°	1.5861
30°	1.5771

Since these figures show a regular variation of 0.0090 for each 5 degrees the figures for intermediate temperatures are interpolated as follows:

TABLE 6

Temperature.	Sp. gr.	Temperature.	Sp. gr.	Temperature.	Sp. gr.
15.0°	1.6041	17.2°	1.6001	19.4°	962
.2°	037	.4°	1.5998	.6°	958
.4°	034	.6°	994	.8°	955
.6°	030	.8°	991	20.0°	951
.8°	027	18.0°	987	.2°	947
16.0°	023	.2°	983	.4°	944
.2°	019	.4°	980	.6°	940
.4°	016	.6°	976	.8°	937
.6°	012	.8°	973	21.0°	933
.8°	009	19.0°	969	.2°	929
17.0°	005	.2°	965	.4°	926

21.6°	922	24.4°	872	27.2°	821
.8°	919	.6°	868	.4°	818
22.0°	915	.8°	865	.6°	814
.2°	911	25.0°	861	.8°	811
.4°	908	.2°	857	28.0°	807
.6°	904	.4°	854	.2°	803
.8°	901	.6°	850	.4°	800
23.0°	897	.8°	847	.6°	796
.2°	893	26.0°	843	.8°	793
.4°	890	.2°	839	29.0°	789
.6°	886	.4°	836	.2°	785
.8°	883	.6°	832	.4°	782
24.0°	879	.8°	829	.6°	778
.2°	875	27.0°	825	.8°	1.5775

Meal.—To determine the lowering of the specific gravity of the carbon tetrachloride by increasing quantities of extracted oil a meal of known oil content (8.82 per cent.) was used. Quantities of this meal were used representing 30-gram portions each of 5.0, 7.5, and 10.0 per cent meals; thus for

5.0 per cent. used 17.0068 grams;
7.5 per cent. used 15.5102 grams;
10.0 per cent. used 34.0135 grams.

These portions were extracted, each with 75 cc. of carbon tetrachloride in Erlenmeyer flasks for 5 minutes at 70°. The filtered extracts showed the following specific gravities:

TABLE 7

Temperatures.	5 per cent.	7.5 per cent.	10 per cent.
15°	1.5900	1.5830	1.5758
20°	1.5810	1.5740	1.5668
25°	1.5720	1.5650	1.5578

The same difference in specific gravity of 0.0018 for each degree of temperature is observed here as was found with the carbon tetrachloride alone, but the decrease in specific gravity due to the increasing quantities of oil is not quite so regular. Thus the decrease due to the oil in 30 grams of a 5 per cent. meal is 0.0141, but in a 10 per cent. meal 0.0283. To test this more carefully a new and more delicate Westphal balance was obtained. The meal used contained 8.20 per cent. of oil. Quantities of this meal were used, representing 40 gram portions each of 5 and 10

per cent. meals, the volume of carbon tetrachloride being increased accordingly to 100 cc. In order to avoid too rapid change in the temperature of the extract during the determination of the specific gravity, the tube containing the extract was placed in a 600 cc. plain beaker filled with clear water to the level of the extract. Different specimens of carbon tetrachloride were used in the two sets of experiments. Corrections for temperature were made on the basis of 1°=0.0018 difference in specific gravity.

TABLE 8

FIVE PER CENT MEAL.

SPECIFIC GRAVITY OF CARBON TETRACHLORIDE AT 26.4°=1.5866.

Extract	Temperature.	Specific gravity.	Calculated to 26.4°.	Difference.
No. 1	26.7°	1.5718	1.5723	0.0143
No. 2	26.8°	1.5716	1.5723	0.0143

TEN PER CENT MEAL.

SPECIFIC GRAVITY OF CARBON TETRACHLORIDE AT 24°=1.5918

Extract	Temperature.	Specific gravity.	Calculated to 24°	Difference.
No. 1	24.0°	1.5632	1.5632	0.0286
No. 2	24.2°	1.5628	1.5632	0.0286

The decrease in specific gravity is, therefore, regular and each per cent. of oil lowers the specific gravity 0.00286 when 40 grams of meal and 100 cc. of carbon tetrachloride are used. The per cent. of oil in a meal is obtained, therefore, by determining at the same temperature the difference between the specific gravity of the carbon tetrachloride and the filtered extract from 40 grams of the meal digested with 100 cc. of the carbon tetrachloride and dividing this difference by 0.00286.

CONSTANT FOR CARBON TETRACHLORIDE.

The difficulty in securing different lots of commercial carbon tetrachloride of exactly the same specific gravity led to experiments with a grade differing widely from that used in the earlier part of the investigation. The following results were obtained:

TABLE 9

Temperature.	Sp. gr. original carbon tetrachloride.	Sp. gr. new carbon tetrachloride.	Difference.
15°	1.6041	1.5903	0.0138
20°	1.5951	1.5813	0.0138
25°	1.5861	1.5723	0.0138

Having determined this difference, 0.0138, in all operations with this new lot corrections should be made by adding the constant to the observed specific gravity and immediate reference could be made to the table of specific gravity of the original carbon tetrachloride. Such a procedure would obviate the necessity of using a thermostat. To test this, 40 grams of meal containing 8.50 per cent. of oil were digested with 100 cc. of the new lot of carbon tetrachloride for 5 minutes at 70°. The extract showed a specific gravity of 1.5572 at 20°; adding the constant 0.0138 this becomes 1.5710, a difference of 0.0241 from the original carbon tetrachloride. Dividing by 0.00286 the per cent. was found to be 8.43.

TIME AND TEMPERATURE EXPERIMENTS.

Some specimens of commercial carbon tetrachloride boil at lower temperatures than that of pure carbon tetrachloride (76.7°). It was deemed advisable, therefore, to determine the rate and completeness of extraction at lower temperatures. In the determinations the per cents. were obtained by the specific gravity method, using a meal whose oil content had previously been found to be 8.82 per cent. The results follow:

TABLE 10

Temperature.	5 min.	10 min.	15 min.	20 min.	30 min.	Standing over night.
25°	7.32	7.50	7.32	7.50	8.00	{ 8.16 { 8.19
*25°	7.50	. . .	{ 8.00 { 7.81	..	7.93	..
40°	7.72	7.72	7.88	..	..	..
50°	7.72	8.35	8.72	..	..	..
60°	8.06	8.06	8.75	..	..	..
	8.08	8.06	8.81	..	..	..

From this table it is evidently impracticable to work at room

*Shaking at intervals.

temperatures. The results show, however, that a complete extraction is obtained in 15 minutes at temperatures 50°–60° C. At such temperatures there is no risk of the commercial carbon tetrachloride boiling.

Confirmation of the accuracy of the method for cottonseed meal is furnished by the following check analyses, for many of which we are indebted to Mr. Jas. B. Pratt, chemist of the Southern Cotton Oil Co., Charlotte, N. C., and Mr. O. L. Spurlin, chemist of the Georgia Cotton Oil Co., Atlanta, Ga.

TABLE 11

		Specific gravity method.	
No.	Soxhlet extraction.	I.	II.
1	8.81	8.72	..
2	6.47	6.44	..
3	6.49	6.59	6.56
4	7.17	7.25	7.27
5	6.47	6.47	..
6	7.17	7.22	7.28
7	7.13	7.19	7.11
8	6.69	6.78	6.69
9	6.27	6.31	6.22
10	6.47	6.56	..
11	7.17	7.06	..
12	6.27	6.35	6.34
13	7.13	7.06	..
14	6.69	6.59	..
15	7.20	7.17	7.23
16	6.70	6.71	..
17	5.96	5.94	..
18	6.40	6.43	..

Meats—Since the per cent. of oil in meats is high and varies between approximately 28 and 36 per cent., it was decided to use 10 grams of meats, instead of 40 as in the case of meal, with 100 cc. of carbon tetrachloride, in order to bring the specific gravity within the limit already worked out for meal, multiplying the per cent. found by four for the actual per cent. of oil in the meats. The use of a wire gauze strainer was unnecessary with meats.

TABLE 12

PER CENT. OF OIL IN UNCOOKED MEATS.

Temperature.	Time.	I.	II.
70°	5	27.12	27.40
70°	15	29.12	29.28
70°	30	29.44	..
60°	15	29.28	..

From these results it is evident that the standard time and temperature of extraction of meal is suited also for extraction of the one-fourth quantity of meats. Experiments using a larger proportion of meats showed low results. Whether this was due to incomplete extraction or to a possible change of the specific gravity curve for such high per cents. was not determined.

TABLE 13.

PER CENT. OF OIL IN COOKED MEATS

Temperature	5 min		10 min		15 min.	
	I.	II.	I.	II.	I.	II
50°	29.84	..	29.72	..	29.60	29.72
60°	29.60	..	29.88	..	30.34	..
70°	29.40	29.88	30.22	30.76	..	..

Again the extraction at 60° for 15 minutes is sufficient.

That the presence of moisture does not affect the extraction was demonstrated by extractions on both raw and cooked meats at 60° C., for 15 minutes, thus:

TABLE 14.

RAW MEATS (moisture 8.95):

	I.	II.
Hydrous	29.28	..
Anhydrous	29.40	29.12

COOKED MEATS (moisture 5.73):

	I.	II.
Hydrous	30.84	..
Anhydrous	31.40	31.00

Hulls.—After transferring 40 grams of hulls to the Erlenmeyer flask it was found necessary to pack the hulls with a glass rod in order to insure complete covering with 100 cc. of carbon tetra-

chloride. No wire gauze strainer was necessary in filtering the extract.

TABLE 15

Temperature.	Time. min.	Per cent. of oil. Soxhlet extraction.	Sp. gr. method.
60°	5	0.30	0.25
60°	10	..	0.32
60°	15	..	0.25
60°	30	..	0.25

It is proposed to extend the investigation to the determination of oil in linseed products, and of fats in feedstuffs, tankage, etc.

University of North Carolina,
Chapel Hill, N. C., October 29, 1908.

THE STABILITY OF ROSIN AT SLIGHTLY ELEVATED TEMPERATURES*

BY CHAS. H. HERTY AND W. S. DICKSON

On heating American rosin to 120°-140° C. in a current of air freed from carbon dioxide, Schwalbe[1] obtained a copious precipitate of barium carbonate by conducting the gases from the flask in which the rosin was heated into a solution of barium hydroxide. He interpreted this as evidence of the decomposition of the abietic acid in the rosin with consequent formation of the hydrocarbon abietene, and pointed out the effect such a decomposition must have upon the melting point and saponification number of rosin.

From evidence obtained during the course of another investigation we were inclined to doubt the accuracy of Schwalbe's interpretation. Accordingly the following investigation was undertaken, the results of which show that rosin which has not been long exposed to the oxygen of the atmosphere can be heated indefinitely at 140° without showing any evidence of the formation of carbon dioxide, provided oxygen and moisture are excluded from the flask in which the rosin is heated.

EXPERIMENTAL

At the outset Schwalbe's experiment was repeated. For heating the rosin a 200cc. Erlenmeyer flask was placed in a beaker containing cottonseed oil. The air entering the flask was freed from carbon dioxide by being drawn through three wash bottles filled with a strong solution of sodium hydroxide. After leaving

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[1] Zeit. angew. Chem, 18, 1852.

the flask the air was passed through a test tube half filled with freshly filtered barium hydroxide solution. Another wash bottle containing a solution of sodium hydroxide was placed between the barium hydroxide tube and the aspirator, a suction pump. A blank experiment with this apparatus showed no precipitation of barium carbonate after drawing air through for one hour. A repetition of Schwalbe's experiment showed a copious precipitation of barium carbonate.

The possibility that this evolution of carbon dioxide might be due to the action of the air upon the heated rosin, aided by the presence of slight traces of spirits of turpentine in the rosin, led to a repetition of the experiment using spirits of turpentine alone instead of rosin. With a specimen of old spirits of turpentine an even heavier precipitation of barium carbonate occurred than with rosin. No question of the splitting off of a carboxyl group could arise here. A specimen of freshly distilled turpentine showed also a precipitation of barium carbonate, but not so marked as with the old specimen.

Having proved that the spirits of turpentine alone was capable of giving the precipitation observed by Schwalbe, a current of steam was passed through molten rosin for eight hours in order to completely remove all spirits of turpentine. Repeating Schwalbe's experiment with this rosin the precipitation was still observed. Evidently the presence of slight traces of spirits of turpentine was not alone responsible for the precipitation observed.

It remained therefore to determine the possible influence of oxygen and of moisture on the formation of carbon dioxide from the molten rosin. Accordingly, the current of air drawn through the flask was freed first from carbon dioxide by sodium hydroxide, then dried by passing through sulphuric acid. A marked precipitation of barium carbonate was again observed. Then moist nitrogen was substituted for air. The nitrogen was prepared by drawing air through three wash bottles filled with an alkaline solution of pyrogallic acid. Again a precipitation of barium carbonate occurred. Finally a current of dry nitrogen was drawn through the flask and after all air had been expelled the rosin was heated

to 140° and kept at this temperature for seven hours without the slightest precipitation in the tube containing barium hydroxide.

The above experiments were carried out on a specimen of freshly distilled rosin from the oleoresin of *Pinus heterophylla* (Cuban Pine). This suggested the possibility that Schwalbe had used a rosin from the oleoresin of *Pinus Palustris* (Longleaf Pine). Accordingly a fresh specimen of rosin was prepared from the oleoresin collected from a single tree of this species. On heating the rosin in dry nitrogen to 140°, again no trace of precipitation was noticeable.

Finally Schwalbe states that his experiment was made upon a sample of commercial American rosin. On heating such a sample in dry nitrogen we found an abundant precipitation of barium carbonate.

Four factors therefore may have entered into the formation of the carbon dioxide observed in Schwalbe's experiment: first, traces of spirits of turpentine in the rosin; second, moisture; third, oxygen in the air conducted through the flask; and fourth, oxygen absorbed either by the oleoresin previous to distillation or by the rosin on standing in the air. The explanation of a probable splitting off of a carboxyl group is demonstrated to be erroneous by using a sample of rosin recently distilled from a fresh specimen of the oleoresin and heating in a current of dry nitrogen.

And yet, paradoxical as it may at first appear, Schwalbe's explanation is even more than true: not in regard to "American rosin", but as applied to the acids of the oleoresin from which rosin is prepared. In order to avoid any elevation of temperature in the preparation of these acids, freed from the other constituents of the oleoresin, a specimen of the oleoresin of *Pinus heterophylla* was dissolved in ether. From this ethereal solution the potassium salts of the acids were precipitated by addition of a saturated water solution of potassium hydroxide. The crystal broth was mixed with glass wool to render it more permeable to an extractive, then thoroughly extracted with ether in a Soxhlet extractor. After removal of the last traces of ether the salts were dissolved in water, the solution acidified with dilute hydrochloric acid, and the precipitated acids washed and dried. On heating a

specimen of these acids in the apparatus described above in a current of dry nitrogen the mass melted at 65°-70°, immediately evolution of carbon dioxide began as shown by the escape of gas bubbles from the molten mass, and the heavy precipitation of barium carbonate.

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